PREPARATION AND EVALUATION OF Al-Ni COMPOSITE POWDERS SUITABLE FOR FLAME SPRAYING

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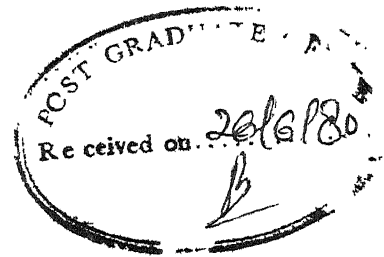## MASTER OF TECHNOLOGY

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to the

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CERTIFICATE

Certified that the work 'Preparation and Evaluation of Al-Ni Composite Powders suitable for Flame Spraying' has been carried out under my supervision and the same has not been submitted elsewhere for a degree.

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ABSTRACT

Al-Ni composite powders were prepared by coating nickel onto aluminium powder by a chemical reduction process known as electroless plating using alkaline baths with sodium hypophosphite as the reducing agent. The effect of various operating variables such as temperature, pH, plating time, bath load etc. were studied to arrive at optimum conditions for obtaining Al-Ni composite powders. Physical and chemical properties of these powders were also evaluated. Attempts were made to prepare wires having an aluminium shell filled with Al-Ni composite powder. Such wires could be useful in flame spraying operations for increasing the bond strength of the sprayed metal coating with the substrate.

CHAPTER 1

INTRODUCTION

Flame spraying is used widely in production for the
application of coatings resistance to oxidation, corrosion,
abrasion erosion and impact. This process is constantly
expanding in many field of activity from the simple reclaiming
of a worn sealing or bearing housing to the design and
manufacturing sphere. In almost all cases of reclaimation
the success of the spray coating depends upon good bond
between the base and sprayed metal. Recently much attention
has been paid for development of materials and processes
producing increased bond strengths.

Molybdenum is widely used as a bond coating or buttering
layer. It has been found that the Al/Ni composite powder
forms an intimate metallic contact with the base metal during
spraying and thereby produces high bond strength. Further
it is reported that the bond strength with Al-Ni composite
powder is much better than with molybdenum due to the exother-
mic reaction between aluminium and nickel.

Al-Ni composite powders are produced by Sherritt
Gordon process which involves the reduction of nickel ions
from ammonical solution by gaseous hydrogen at a pressure of
about 350 psi and a temperature of $180-200^{\circ}C$ onto the suspended

aluminium powder particles. However, this technology involving high temperature and pressures is costly and complicated.

The present study was undertaken in order to study the feasiblity of producing aluminium-nickel composite powder by electroless plating of nickel over aluminium powder. The possibility of producing wires using these Al/Ni composite powders and which could be useful for flame spraying by wire guns, was also investigated.

CHAPTER 2

LITERATURE REVIEW

2.1 METAL SPRAYED COATING

Metal spraying is one of several methods of depositing coatings and the apparatus used consists of spray gun similar to those used in paint spraying. This type of coating may be applied from a base material in wire or powder form and the flame may be generated by a combustion of gases, plasma arc or electric arc. Each system offers separate advantages and as much care should be taken in the selection of the system of deposition as in the selection of material to be deposited. An extensive literature about metal spraying processes is described in the text book by Ballard[1].

Metals commonly sprayed by the oxyacetylene wire gun include aluminium, nickel, copper, zinc, bronze, lead, molybdenum, tin etc., as well as low carbon, high carbon, and stainless steels. This type of gun is used successfully as a maintenance-repair tool and for the salvaging of shafts, rolls, bearing housings, pump rods and other parts that require a buildup of worn surfaces. Another gun is the oxy-acetylene powder gun mainly for hard-facing applications. The gun can spray metal powder in place or over almost any contoured surface. Also hard, corrosion-resistance alloys like nickel base or cobalt base containing chromium, boron, and silicon can be sprayed by the powder gun.

2.1.1 Advantages of Metal Spraying:

The advantages of metal spraying over other metal processes are that:

1. Metal spraying is not limited to any one metal or group of metals. Any metal or alloy obtainable in wire or powder form can be sprayed.

2. The thickness of the coating can be varied at will and selected to suit any requirement.

3. There is no limit to the size of the article to be coated.

4. Although deposits are applied in a molten state, the base material undergoes only a small rise in temperature.

5. The process does not cause any distortion, embrittlement or loss of strength in the base metal.

6. The inherent nature of metal spraying is such that the sprayed coating has microporosity. In many applications this has an advantage as it could retain oil which could provide lubrication during start up period and thereby drastically reduce component wear.

7. In many cases it is possible to deposit a coating which has higher hardness and better corrosion and wear resistance than the base material. Thus the sprayed component could outlast the original component.

2.1.2 Spraying Materials:

Flame spraying in the initial stages of its commercial development was used mostly for the spraying of various metals and was often referred to as metallizing. However now a days different spraying techniques are there which extend to a much wider group of materials, including higher melting point or refractory metals, ceramic, cermets and the like. The following table lists a number of materials and their various combinations which can be sprayed.

Metals	Oxides	Carbides of	Pre-alloyed Powders	Misc. materials
Aluminium	Alumina	Boron	Brass	Nitrides
Chromium	Ceric oxide	Chromium	Bronze	Silicides
Copper	Chromium	Tantalum	Hard-facing alloys	Borides
Gold	Oxides blended with Ni	Titanium	Nickel aluminide	Spinels
Iron		Tungsten	Nickel-chrome alloys	
Moly-bdenum	Aluminide	Zirconium	Stainless steel	
Nickel	Titania			
Silver	Zirconia			
Tungsten	Nickel oxide			
Titanium				
Zirconium				
Platinum				
Cobalt				
Niobium				
Tantalum				

The table includes a number of materials containing Nickel-aluminide as one component. Sheppard[2] reported that this compound which is a mixture of Ni/Al and Ni$_3$Al, was produced by an exothermic reaction between nickel and Al combined together in the original powder which consists of Aluminium particles coated with nickel in the ratio of 20 pct. Al to 80 pct. Ni. The heat reaction within the particles increases their temperature during flight and it was claimed that sufficient heat continues to be generated on the surface after the impact of a spraying particle with substrate to produce a metallurgical bond. In this process the substrates need no longer be shot or grit blasted, which is an advantage when coating thin sections. A bright surface is a satisfactory basis for the adhesion of sprayed layer. The various graded refractory coating containing nickel-aluminide have proved useful materials. These have shown better substrate bonding and more superior thermal shock properties than the refractory material alone.

According to Bell[3], in order to achieve a high deposit density and strong mechanical bond, a close control of particle size was necessary. For example, coarse particles do not always melt completely and too many will produce a poor coating. On the other hand, undersize particles or fines tend to vaporise. The size of particles will vary from 5-100 μm, the samller size ranges produced by powder systems and the

large size by rod/wire spraying. Apart from the condition of the particle, the structure of coating also depends on the velocity of the particles, the denser coating being produced by the high velocity systems typically;

Powder - combustion flame   50 ms^{-1} particle velocity

Powder - Plasma flame   140 ms^{-1} particle velocity

Rod   - Combustion flame 120 ms^{-1} particle velocity

Rod   - Plasma flame   240 ms^{-1} particle velocity

Thus rod deposits tend to be less porous.

2.1.3 Applications of Spray Coating:

Metal spraying is used widely in production for the application of coatings resistance to oxidation, corrosion, abrasion erosion and impact. The majority of metal spray coatings fall under one of the following broad classification.

1. Wear resistance.

2. Heat and oxidation resistance.

3. Atmospheric and sea corrosion resistance.

4. Chemical corrosion resistance.

5. Electrical conductivity or resistivity.

6. Abradability.

7. Restoration of dimensions.

8

Sheppard[4] outlined the characteristics of flame sprayed coatings currently being used by manufactures in a variety of industries. The following table gives an indication of the diversity and scope of flame sprayed coatings.

Characteristics	Typical appliances
Wear resistance	Synchronization rings, gear cones, piston rings, shift forks, roll neckes, plug gauges.
Abradability	Seal areas.
Electrical conduction	Capacitors, varistors, resistors, heating surfaces.
Electrical insulation	Nuclear spark plugs, heater tubes, hand tools.
Solderability	Capacitors, radiators, resistors, thermoglass.
Thermal barrier	Furnace fixtures, rocket throats, Wangel engine rotors, re-entry shields.
Thermal conduction	Stainless steel saucepans.
Spark prevention	Gas cylinders, pit props.
Cavitation resistance	Diesel liners, ship stabilizers.
Resistance to wetting	Welding fixtures, welding strip.
Corrosion resistance	Any component subject to atmospheric, chemical, or elevated temperature corrosion.
Restoration of dimension	Bearing journals, press fits, bearing housings.

2.2 COMPOSITE POWDERS

Many material scientists believe that the most specta-
cular pieces of advanced technology of today's will be built
by the composite materials-like supersonic and hypersonic
aeroplanes, rockets, high-pressure apparatus, spacecraft,
satellites, motor car bodies, deep diving submarines, chemical
equipment, new source of electric power and so on. The term
composite powder refers to a powder each particle of which
consists of two or more phases, usually in the form a coatings
on a central core. For the core particle and the coating a
wide range of metals and nonmetals has been found suitable such
as - Metal/metal (Ni/Al or Ni/Cr), metal/metalloids (Ni/P or
Ni/Si), metal/carbides (Ni/WC), metal/alloys (Ni/steel),
metal/oxides (Ni/Al$_2$O$_3$), metal/nonmetal (Ni/graphite), metal/
mineral (Ni/CaF$_2$), metal/nitrides (Ni/BN), metal/fibrous
materials (Ni/glass, Ni/asbestos), etc.

Despite the fact that composite powders are relatively
new materials, many papers have published suggesting possible
application in various field. The advantage of composite powders
lies in overcoming many of the problems associated with the
compacting and sintering of powder mixtures. It has reported
by Lund[5] that the components in the composite powder compacts
are better dispersed than can be obtained in conventional
mixed powder compacts. The achievement of more rapid alloying
by diffusion in composite powder compacts has been attributed

to better mechanical dispersion of the components, greater protection of reactive elements against oxidation or other reactions at elevated temperatures, and more intimate contact between the components over a greater surface area than is possible in mixed powder compacts. These powders have a common large surface area which affects the kinetics of the reaction between core and coatings, and they behave-despite their heterogeneous nature-as an homogeneous mass during the storing and handling. The powders can either be consolidated by pressing and roll compacting, or can be applied by flame and plasma spraying.

In special cases a composite powder has been designed such that the core and coating constitute two components of a highly exothermic reaction. For example Ni/Al (82-18) composite powder developed by Metco. Aluminium is a core and nickel as a shell. So in this state the aluminium is protected from atmospheric oxidation and the composite powder can be heated without fear of losing aluminium as Al_2O_3. As the Ni/Al composite powder is heated, a sudden very vigorous, exothermic reaction sets in and the nickel and aluminium react to form Ni-aluminide[2]. The literature gives 117-284 KJ/mol as the enthalpy of formation depending on which Ni-aluminide intermetallic compound is formed and Hermanek[6] reported a temperature of $3185^{\circ}C$ for the superheated liquid particles of Ni/Al derived from this reaction.

2.2.1 Preparation of Composite Powders:

Medding B. et al.[7] have reported different processes
for the preparation of composite coated powders, such as the
thermal decomposition of metal carbonyls, the displacement
reactions involving surface diffusion, the hydrogen reduction
of volatile compounds and liquid-phase deposition involving
electroless and Sherritt-Gordon processes. At present the
composite powders are produced on commercial scale by
Sherritt-Gordon[8] process, which involves the reduction of
metal ions from ammonical solution by hydrogen, at elevated
temperature and high pressures on to the suspended core powder
particles. The core powder (metallic/non metallic) to be
coated is suspended by mechanical agitation in an ammonical
solution of nickel ammonium sulphate. The solution is then
reduced to nickel by gaseous hydrogen at a pressure of about
350 psig and a temperature of 350-400°F in an autoclave. The
essential reaction occuring can be represented as—

$$NiSO_4 + 2NH_3 + H_2 \longrightarrow Ni + (NH_4)_2SO_4$$
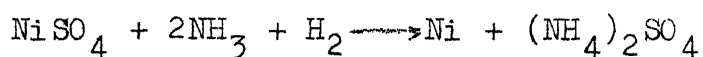
Upon reduction the nickel nucleates preferentially on the
suspended core particles and coats them uniformly with a
layer of nickel. The size of the core particle is limited
by the need to maintain the particles in suspension by agitation,
depending on density, particles upto several hundred microns
can be coated.

Kunda[9] described over 50 composite powders utilizing many metals, oxides, and compounds as cores with coating of Ni, Co, Cu, Ag and Mo either separately, simultaneously or alternatively in multiple layers.

2.2.2 Applications of Composite Powders in Thermal Spraying:

Composite powders consisting of a metal or alloy coating on a metal or non-metal core possess certain advantages in thermal spraying applications. These may be summarized as follows:

1. The heterogeneous nature of the individual particles combined with the homogeneous nature of the bulk powder permits unusual material combinations in the sprayed deposit.

2. The components of the composite powder can be such that they will undergo some desirable reaction with each other during spraying.

3. The metallic coating ensures good particle to substrate and interparticle bonding in the sprayed deposit.

4. The good interparticle bonding results in good deposit efficiencies for metal coated composite powders.

5. The metallic coating protects the core from oxidation during spraying. In the case of carbide powders this amounts to preventing decarburization.

6. Due to the protection of the coating, finer metal coated carbide composite powders can be sprayed without

decarburization, thus taking advantage of the finer
powders for better surface finishes.

7. In common with other thermal spray materials, composite
powders are ideally suited to maintenance and repair
work to which they bring a wider combination of materials.

Composite powders in thermal spray applications include
Ni/Al for bond coating, Ni/Graphite and NiCr/Diatomite for
abradable seals, Co/WC, Ni/WTiC$_2$ and NiCr/Cr$_3$C$_2$ for wear
resistance applications and Ni/CaF$_2$ and NiO/CaF$_2$ for low
friction seals.

2.3 ELECTROLESS PLATING

Electroless plating was discovered by A. Branner and
G. Riddell[10,11] in 1944. A number of publications and patents
have appeared since that time. More recently, the name
'autocatalytic plating' has been preferred which may be defined
as 'deposition of a metallic coating by a controlled chemical
reduction that is catalyzed by the metal or alloy being
deposited.' Since autocatalytic plating takes place on a
catalytic surface, it is evident that once deposition is
initiated the metal deposited must itself be catalytic if
deposition is to continue. Metals that catalyze the plating
are Fe, Co, Ni, Al, Pd, Cu and sometime Ag and Au. Other
non-catalytic metals and also non-metals can be plated, if
their surfaces can be made catalytic by applying a strongly
catalytic metal on the surface by chemical or mechanical means.

2.3.1 Principle of Electroless Plating:

Electroless plating involves reduction of a metal salt to the metal with simultaneous oxidation of a chemical compound called the reducing agent. To prevent, or at least minimize the tendency for oxidation-reduction reaction to take place throughout the plating solution, electroless plating solutions are formulated so that the concentration of metal salt, and reducing agent, and the pH are such that metal reduction does not occur readily. This being so, the work would not be plated either, this problem is overcome by the use of catalysts which localize the plating reaction to desired surface only. If the metal being deposited serves as a catalyst, deposits may be built up as thick as desired.

A simplified equation that describe the formation of electroless Ni deposition is:

$$NiSO_4 + NaH_2PO_2 + H_2O \xrightarrow[\text{catalyst}]{\text{Heat}} Ni + NaH_2PO_3 + H_2SO_4$$

or

$$Ni^{++} + 2H_2PO_2^- + 2H_2O \longrightarrow Ni + 2HPO_3^{--} + 4H^+ + H_2$$
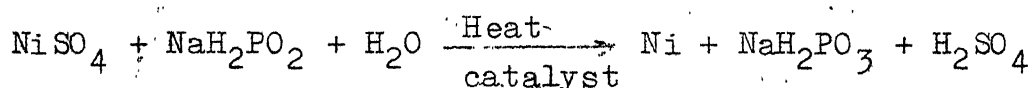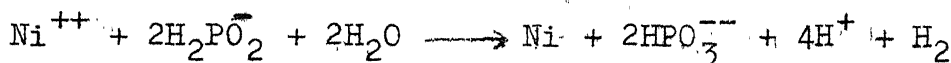
(hypophosphite ion)   (phosphite ion)

The complete mechanism of this reaction has been the subject of considerable study, at present the most likely route seems to be Lukas R.M.[12] hydride mechanism as follows:

$$H_2PO_2^- \xrightarrow{\text{catalyst}} PO_2^- + 2H \text{ (catalyst)} \qquad \dots(1)$$

$$PO_2^- + H_2O \longrightarrow H^+ + HPO_3^{--} \qquad \qquad ...(2)$$

$$Ni^{++} + 2H \text{ (catalyst)} \longrightarrow Ni + 2H^+ \qquad \qquad ...(3)$$

$$H_2PO_2^- + H \text{ (catalyst)} \longrightarrow H_2O + OH^- + P \qquad \qquad ...(4)$$

$$2H \text{ (catalyst)} \longrightarrow H_2 \qquad \qquad ...(5)$$
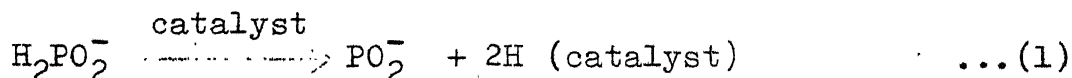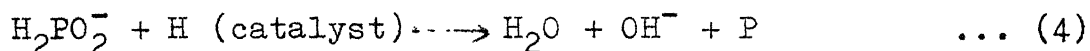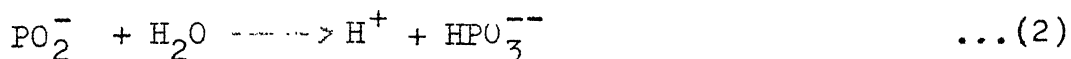
Reaction (1) expresses the fact that hypophosphite anions are hydrogenated catalytically to metaphosphite ions. This abstracted hydrogen, either in the atomic or in any 'activated form' is adsorbed on the catalyst (broadly considered as hydride ion). Reaction (2) shows that the metaphosphite ion reacts with water to form orthophosphite anion. This equation indicates the formation of hydrogen ion (H^+), which explains the pH dependency of reactions (1) and (2). Both this reactions are of course occur simultaneously. Reaction (3) indicates the reduction of nickel ion to metallic nickel by active hydrogen atoms adsorbed on the catalytic surface, with formation of hydrogen cations. Reaction (4) represents the dehydration - reduction of hypophosphite to active phosphorus, which combine with Ni to form Ni-P alloy. Reaction (5) represents the formation of molecular hydrogen gas.

It can be seen that reactions (1) and (2) together are favoured by a high pH value. This is also true for reaction (3), because H^+ ions are formed on the right hand side. On

the other hand, reaction (4) must be slowed down by a high
pH as one of its products is the hydroxyl (OH^-) ion.

In acid solution, there is a further reaction to be
considered:

$$Ni + 2H^+ \longrightarrow Ni^{++} + H_2 \qquad \qquad \ldots (6)$$

This reaction expresses the fact that the deposited nickel
is dissolved, with evolution of hydrogen gas, when the pH
is sufficiently low.

Considering reaction (3) it can be seen that in a
batch operation the solution will become more and more
acidic as the reaction proceeds, untill the rate of reaction
(6) becomes equal to that of the nickel alloy deposition, and
the latter will eventually stop. In order to allow the
deposition reaction to proceed continuously or to completion,
it is necessary to keep the pH substantially constant. This
can be achieved by periodic or continuous addition of a
neutralizing chemicals i.e. of hydroxyl ions. It can also be
promoted by incorporating a buffer in the bath composition
(like citric, acetate or malic anions).

2.3.2 Bath Compositions:

Two distinctly different types of electroless nickel
baths are in use, acid baths at pH 4-7/and alkaline baths at
8-11. Commercially the acid baths have been the more important.

Gutzeit[13] has given the role of various constituents in electroless Ni solutions as follows:

1. Nickel salt: supplies the applied source of metallic nickel. The choice of Ni salt is not too critical.

2. Hypophosphite: to supply, by catalytic dehydrogenation, active hydrogen atom for reducing the Ni^{++} to the metal. This is also the source of phosphorus in the deposit.

3. Chelating agents: usually a hydroxycarboxylic acid or salt, such as hydroxyacetic, citric or malic etc. to form complex or chelate the nickel ions in order to prevent nickel phosphite precipitation.

4. Buffering agent: usually, the same as the chelating agent, though acetates and propionates are also used to control the rapid decrease in pH of operating baths.

5. Stabilizers: to prevent solution decomposition by adsorbing on active nuclei which meight form in the bath. Sulfur compounds like thiourea, PbS, Sn sulfides etc. are generally used.

6. Wetting agent: to promote wetting of parts being plated, usually anionic type.

7. Brightener: to get bright coating, some organic agents are added.

(i) Acid Baths:

The acid baths are more widely used in commercial installation than alkaline baths, because these are more stable than alkaline and are easier to control, regenerable and usually provide a higher plat rate. Essentially acid baths contain Ni salt, a hypophosphite salt, and a buffer, some solutions also contain chelating agents. Some of the acid baths, their composition and operating conditions are given below:

Constituent or condition	Bath 1	Bath 2	Bath 3
Nickel chloride	30 gm/l	-	-
Nickel sulfate	-	21 gm/l	20 gm/l
Sodium hypophosphite	10 gm/l	24 gm/l	27 gm/l
Sodium succinate	-	-	16 gm/l
Sodium hydroxyacetate	50 gm/l	-	-
Lactic acid (80 pct.)	-	34 ml	-
Propionic acid (100 pct.)	-	2.2 ml	-
pH	4-6	4.3-4.6	4.5-5.5
Temperature	90°C(190°F)	203°F	$200-210^\circ$F
Plating rate (mil/hr)	0.5	1.0	1.0

1) US Patent 2,532,283 (2) US Patent 2,822,393 and 2,822,294
(3) US Patent 2,658,841 and 2,658,842.

(ii) <u>Alkaline Baths</u>:

In practice these baths plate at a slow rate and are reported to be uneconomical because of the rapid loss of ammonia at the high temperature of operation. Certain advantages are claimed for the alkaline bath.

i) It is cheaper to operate than acid baths.

ii) Less critical control is required. Adjustment of pH is made with NH_4OH (50 pct.). A definite colour change blue to green is visible when the pH is too low, indicating the need for addition of NH_4OH.

iii) The deposits are whiter and brighten than from the acid baths.

Following table gives the compositions and operating conditions for different alkaline electroless baths.

Constituent or condition	Bath 4	Bath 5	Bath 6
Nickel chloride	30 gm/l	–	45 g/l
Nickel sulphate	–	25 g/l	–
Sodium hypophosphite	10 gm/l	25 g/l	11 g/l
Sodium citrate	100 gm/l	–	100 g/l
Ammonium chloride	50 gm/l	–	50 g/l
Sodium pyrophosphate	–	50	–
pH	8–10	10–11	8.5–10
Temperature	90°C(190°F)	150–170°F	195–205°F
Plating rate		0.6 mil/hr	0.4 mil/hr

4. US Patent 2,915,406

5. Schwartz M., Proc. Am. Electropl. Soc. 47, 176 (1966)

2.3.3 Applications of Electroless Nickel Plating:

The deposit obtained from an electroless Ni solution
is usually an alloy containing from 4-15 pct. phosphorus and
is quite hard. Also the deposit is not dependent on current
distribution, it is uniform in thickness, regardless of the
shape or size of the plated surface. Electroless Ni deposits
may be applied to provide the basis metal with resistance to
corrosion or wear, or for the build up of worn areas. Typical
application of electroless nickel are:

1. Plating applied for corrosion resistance.

    Valve body, Reactor vessel, pressure vessel, Printing

    rolls, tubular shaft and so on.

2. Plating applied for wear resistance.

    Centrifugal pump, Printing press bed, Hydrolic pistons,

    Stator and Motor blades ,spray nozzeles etc.

3. Plating applied for buildup of worn areas.

    Carburized gear, Splined shaft, connecting arm.

Thus numerous applications have been developed for
electroless nickel deposits in the past and have been found
ready acceptance in fields such as missiles, rocketry, oil
refinaries, chemical plant and equipments and many other
electrical applications. Due to hardness, corrosion resistance,
and uniform deposit thickness of electroless Ni, the coating

has also found wide usage in the field of plating moving parts
for pump , compressors and other hydraulic equipment. In-
fact, electroless nickel should be considered whenever there
is a need for a hard, smooth and corrosion resistance coating.
In most cases the deposit thickness to protect parts from
mild corrosion nearly 0.5 mil and for severe corrosion upto
3.0 mils are needed. A coating of electroless nickel can be
utilized to inhibit stress corrosion cracking of stainless
steel and titanium alloys. This coating is also frequently
plated on aluminum parts to provide a solderable surface.

The low coercivity of electroless nickel-phosphorus
and nickel-cobalt-phosphorus alloys, and the relationship of
their remanent and saturation magnetic forces, can be utilized
for fast-switching memory devices in computers. The
metallization of non-conducting surfaces, which has a vital
role in the plating of plastic, is another widespread use for
electroless nickel deposit.

It is true that now a days chemically deposited nickel
(electroless nickel) is firmly established as a production
process for hard facing and protecting most metals and alloys.
But it is more expensive than electroplating because chemical
reducing agents are much more expensive source of electrons
than the electric current. Hence high capital outlay and the

specified nature of the process still restrict its use by the individual companies. Due to its superior corrosion resistance it has been found to be very worth while, particularly in the treatment of parts which are subjected to very severe conditions.

CHAPTER 3

EXPERIMENTAL TECHNIQUES

3.1 PRETREATMENT OF ALUMINIUM POWDERS:

Aluminium powder was obtained from Robert Johnson (India) company and was screened through -325 mesh. This as received powder was first tried for electroless plating of nickel. It was however, noticed that practically no deposition took place. Therefore it became evident that pretreatment of aluminium powder was necessary. Several pretreatment solutions and procedures are described in the literature for aluminium and its alloys. The following pretreatment was found to be most suitable.

1. **Alkali Treatment** (for degreasing the powder)

200 c.c. of water was added to approximately 5 gm of the aluminium powder contained in 1 litre measuring cylinder. To this 10 c.c. of 1 pct. sodium hydroxide solution was added slowly with stirring for 3 to 4 minutes. Aluminium powder was then allowed to settle.

2. **Rinsing**

Alkali solution was decanted and the powder was washed 2 to 3 times with water in order to remove the traces of alkali solution, leaving about 200 c.c. of water after final rinsing.

3. <u>Acid Treatment</u>

5 c.c. of 5 pct. sulfuric acid and 2 c.c. of 1 pct. hydrofloric acid were added slowly to the above with stirring and after 2/3 minutes acid was decanted.

4. <u>Washing</u>

The powder was washed thoroughly 2/3 times with water to remove the acid completely. Final washing was done with distilled water.

5. <u>Drying</u>

After removing water by filtration, the powder was air dried in oven at 80°C for four to five hours. This resulted in slight agglomerated mass/which was ground using glass pastel and mortar and then screened through -325 mesh. The screened powder was stored in air tight container for subsequent plating.

3.2 <u>NICKEL COATING OF ALUMINIUM:</u>

The general experimental set up used in all plating work is shown in Figure 3.1. The following steps were involved for coating of aluminium powder.

1. Constant temperature bath was heated for nearly an hour to get the temperature 91 ± 1°C, meantime the alkaline bath solution of given composition (Table 3.1) was prepared and the pH was adjusted using 1:1 ammonia solution.

2. Prepared solution (100 c.c.) was taken in a double walled glass vessel and hot oil at $91 \pm 1^{\circ}C$ from a constant temperature bath was circulated. When the temperature of the plating solution reached $90 \pm 1^{\circ}C$, 2 gm of pretreated aluminium powder was put into it and stirred gently. Reaction was allowed to continue for a fixed time, i.e. 5, 10, 15, 30 minutes.

3. The coated powder along with the plating solution was taken out in a clean beaker. It was then filtered out using sintered glass crucible (G4). The coated powder was washed 2/3 times with distilled water.

4. The crucible was dried in an air drying oven at approxmately $90^{\circ}C$ for four to five hours and thereafter cooled in a desicator. The powder so obtained was stored in a air tight bottle and kept in a desicator for subsequent analysis.

3.3 CHEMICAL ANALYSIS OF NICKEL COATED ALUMINIUM POWDERS:

The composite powder obtained by the electroless plating under various operating conditions were analysed for nickel and phosphorus.

3.3.1 Determination of Wt. pct. Nickel:

The standard gravimetric method[14] (dimethylglyoxime method) was used to determine the nickel. Around 0.2 gm

(accurately weighed) of the coated powder was dissolved in 50 ml of 1:1 nitric acid in a beaker and then transferred to a standard 100 ml volumetric flask and made upto the mark. 10 ml of this solution was taken in a beaker and 10 ml of 5 pct. citric acid solution (to supress the precipitation of aluminium in ammonical medium) was added. Then solution was warmed to approximately $50^{\circ}C$. To this solution nearly 20 ml of 1 pct. ammonical dimethylglyoxime solution was added immediately followed by dilute ammonia solution until the liquid was slightly alkaline. The solution was heated at $60^{\circ}C$ for an hour. The precipitate was filtered out using a weighed sintered glass crucible (G4) and washed well 2/3 times with distilled water. The precipitate was dried at $110-120^{\circ}C$ for two hours, cooled in a desicator and weighed as nickel-dimethylglyoxime (Ni-dmg).

Calculation:

$$\text{Wt. pct. Ni} = \frac{\text{Wt. of Ni-dmg} \times 0.2032}{\text{Wt. of sample taken}} \times 100$$

$$[1 \text{ gm Ni-dmg} = 0.2032 \text{ gm Ni}]$$

3.3.2 Determination of Phosphorus

From the literature it has been found that electroless nickel obtained from plating baths using sodium hypophosphite as the reducer contains phosphorus. Phosphorus was determined gravimetricaly[15] (Mg-Phosphate precipitation)

using the following procedure.

10 ml solution of coated powder dissolved in 50 pct. HNO_3 (as used in Ni analysis) was taken in a beaker and 10 ml solution of 5 pct. citric acid was mixed with it and diluted to 100 ml. 5 gm of ammonium chloride was added into the beaker and shaken well. Few drops of methyl red indicator were added to ensure slight acidity of the solution.

Now 25 ml of magnesia$^\varphi$ mixture added slowly, followed by the addition of 1:1 ammonium hydroxide with constant stirring until precipitation was complete. Few drops of phenolph-thalein were added into the solution. More NH_4OH (1:1) was added dropwise untill the solution was alkaline to that indicator. 15 ml excess of ammonia solution was added. This was then kept overnight. The precipitate formed was washed with 1:20 NH_4OH solution and filtered through filter paper. This precipitate was dissolved in 1:1 HCl (nearly 10 ml) and diluted to 100 ml. 2 ml of magnesia mixture and then 1:1 NH_4OH was added dropwise (added 10 ml excess) and kept it for four hours. The precipitate so obtained was filtered through ashless (Watman 42) filter paper and washed with 1:20 NH_4OH until the wash water showed no trace of chloride ions. This filter paper was ignited gently in a
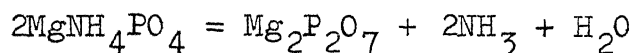

φ Magnesia mixture: Dissolved 55 gm of $MgCl_2 \cdot 6H_2O$ and 140 gm of NH_4Cl in 500 ml water and 130.5 ml concentrated ammonia solution was added, mixed well and diluted to one litre.

weighed porcelain crucible and the temperature of the muffle furnace was gradually raised upto 800-900°C and heated for four hours. The crucible was cooled in a desicator and weighed as magnesium pyrophosphate $[Mg_2P_2O_7]$.

Calculation: The precipitation reaction is:

$$H_3PO_4 + MgCl_2 + 3NH_4OH = MgNH_4PO_4 + 2NH_4Cl + 3H_2O$$
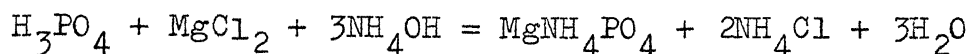
On ignition the decomposition is:

$$2MgNH_4PO_4 = Mg_2P_2O_7 + 2NH_3 + H_2O$$

$$Wt. \ pct. \ P = \frac{wt. \ of \ ppt. \ obtained \ x \ 0.2783}{wt. \ of \ sample \ taken} \ x \ 100$$

3.4 PHYSICAL PROPERTIES OF NICKEL COATED ALUMINIUM POWDERS:

A perfect and complete characterization of metallic powder presents difficulties - due to the large number of variables involved, the complexity of the properties involved and so on. So some of the important properties like particle size, apparent density, flow rate, angle of repose etc. were studied. Further X-ray and thermal analysis of few powders were also made.

3.4.1 Particle Size:

The determination of particle size and distribution of powdered materials is an important factor. Many properties like porosity, density of compact, mould strength, flow and

mixing properties, solubility and reactivity are all affected by particle size.

There are various methods of particle size determination like screen analysis, microscopic analysis, sedimentation methods, elutriation methods and others. In the present work optical microscopy method was used. Since the composite powder after coating got somewhat agglomerated, suitable dispersing media was tried. Amyl alcohol was found to be the best. Little amount of powder was added into amyl alcohol, shaked well and few drops of suspension were taken on a glass slide, dried and examined under a metallurgical microscope for particle size measurement. Crossections of few powders were tried.

3.4.2 Flow Rate and Apparent Density Measurement
(1) Flow Rate:

ASTM standard No. B213-46T, powder flowmeter (sketch in figure 3.2) was used to determine the flow rate. Fixed amount of the coated powder was taken in the funnel and allowed to have a gravity fall in the density cup, kept at a distance of one inch from the bottom of the funnel. The time taken for a weighed sample of powder to flow was noted in seconds and the flow rate was expressed in sec/50 gms.

(2). <u>Apparent Density</u>:

The apparent density of a powder is the weight of
a unit volume, including all voids between particles, internal
porosity etc. The bottom hole of the funnel (Fig. 3.2) was
closed with the finger. The funnel was filled completely
with the composite powder. The powder was allowed to fall
from the funnel into the density cup, placed at a distance
of one inch from the bottom of the funnel, by removing the
finger. The density cup was completely filled with the
powder and the powder levelled off flush with the top of
the cup and weighed the cup's contents. Volume of the cup
was known.

$$\text{Apparent density} = \frac{\text{wt. of powder in the cup (gms)}}{\text{volume of the cup (c.c.)}}$$

3.4.3 <u>Angle of Repose Measurement</u>:

Angle of repose is an important property to describe
the flow behaviour of powder i.e. flowability of powders,
and is defined as the constant angle to the horizontal of a
conical pile of the material. This pile was produced and
measured with the apparatus shown in figure 3.3. The cylinder
was placed so that it just rest on the circular base and
was filled with composite powder. Then the cylinder was
raized a few millimeter so that the powder spilled out.
When the flow stopped the cylinder was gently removed and the

height of cone was measured. The angle of repose was calculated from.

$$\tan \theta \ = \ h/r$$

h = height of the pile (cm)

r = radius of base (cm)

3.4.4 Compressibility of Composite Powder:

Compressibility of powder is a measure of flowability of powder. Measuring cylinder of 25 ml capacity was taken and filled upto the mark with composite powder. Then it was placed on a vibrator for 3 minutes and new volume of the powder was measured. Compressibility was obtained by substracting this volume from the initial volume and expressed as percentage of initial volume.

3.4.5 Differential Thermal and Thermogravimetric Analysis:

DTA and TGA studies of composite Al-Ni powder was done to determine the changes occuring during heating. Fixed amount of powder was taken in ceramic crucible and was analysed using 'DERIVETOGRAPH' (MOM Hungarian Optical Works, Budapest) at the heating rate of $10^{\circ}C/min$, sensitivity 1/10 and upto temperature of $1000^{\circ}C$ in ordinary air atmosphere. Three samples - aluminium, nickel coated aluminium and recoated nickel powder under identical conditions were studied.

3.4.6 X-ray Powder Diffraction Analysis:

For qualitative analysis of the composite powder, X-ray powder diffraction was done using a GE XRD-5 Diffractometer. X-ray diffraction patterns of some samples were obtained with CuK_{α} radiation with nickel filter at 1000 cps, scanning rate of 2°/min. Tube voltage was 35 KV and current was 15 mAmp. Powder obtained under optimum condition was heated to $650^{o}C$ for 1 hour in air furnace and then scanned for X-ray diffraction.

3.4.7 Preparation of Wire Using Al-Ni Composite Powder:

A hole of 5/32" (3.96 mm) diameter and a length 2" (5.08 cm) was made in aluminium rod of diameter 1/4" (6.35 mm) and Al-Ni composite powder was filled inside the cavity with tapping. The mouth of cavity was closed using a screwed cap. The rod so filled was then swaged to 1/8" (3.17 mm) diameter.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effect of Plating Variables

To study the effect of plating variables e.g.
temperature, pH, plating time, bath load etc., the Al-Ni
composite powders were prepared under different plating
conditions.

4.1.1 Effect of Plating Time

The amount of nickel in the composite powder increased
with increasing plating time at all pH, temperature and bath
loads (Table 4.1 to 4.3, Fig. 4.1 to 4.3). The results also
showed that the deposition rate of nickel decreased with
increasing plating time at all pH, temperature and bath
loads. Further it was observed that in all cases the curve
became flattened after the plating period of 30 minutes.
Using this optimum plating time subsequent powders were
prepared.

4.1.2 Effect of pH

As the bath pH increased, the rate of nickel deposition
and the amount of nickel in the composite powder increased
(Fig. 4.4, Table 4.1, 4.2). On increasing pH from 8.0 to 9.0
there was a marked increase in the amount of nickel in the

composite powder. Increasing pH beyond 9.0, although increased the amount of nickel in the composite powder but the effect was not so pronounced. Moreover, it was practically very difficult to maintain pH higher than 9.0 and baths tend to become unstable at pH higher than 10. Therefore for subsequent work solution pH was maintained at 9.0.

4.1.3 Effect of Temperature

Decreasing bath temperature from 90°C to 85°C decreased the rate of nickel deposition and the amount of nickel in the composite powder (Table 4.2, 4.3). Whereas it was difficult to run the bath at temperature higher than 90°C because of increased evaporation losses. Therefore optimum temperature for preparing nickel composite powder appeared to be 90°C.

4.1.4 Effect of Bath Load

Increasing the bath load of aluminium powder from 20 gm/l to 40 gm/l decreased rate of nickel deposition and the amount of nickel in the composite powder (Table 4.2, 4.3). From this it would appear that decreasing the bath load below 20 gm/l would increase the amount of nickel in the composite powder. However, bath loads lower than this will decrease the yield substantially and also decrease the amount of

nickel recovered from the plating bath. Therefore in
subsequent work bath load of aluminium particles was kept
at 20 gm/l.

4.1.5 Effect of Bath Variables on Phosphorus Content in Composite Powder

Phosphorus analysis were done only for few samples.
It was observed that the results were not consistent due to
experimental errors since the powder from the same bath
gave difforent results. However, the range of phosphorus
content in the composite was found to be 4 to 11 percent.

4.2 Physical Properties of Al-Ni Composite Powder

Several physical properties of the composite powders
e.g. particle shape and size distribution, flow rate, apparent
density, angle of repose, compressibility etc. were
determined.

4.2.1 Particle Shape and Size Distribution

The uncoated aluminium powder examined under optical
microscope showed that the particles were angular and irregular
in shape (Fig. 4.6) having an average particle size of
21.04 μm. Whereas the nickel coated aluminium powder produced
at pH 9, time 30 min, temperature 90°C, bath load 20 gm/l
was not so angular (Fig. 4.7) and had a average particle
size of 23.8 μm. Since many particles of composite powder

were agglomerated (Fig. 4.7) all bigger size particles were discarded during the above particle size measurement. The nickel was coated almost uniformly over the aluminium powder (Fig. 4.8). The average thickness of nickel shell was estimated to be about 3-4 μm which matched well with the estimates based on average particle size measurement of coated and aluminium powders.

The particle size distribution (Fig. 4.5) showed that most of the uncoated aluminium particles were within the range of 15 to 20 μm. Whereas for coated powder particles this range was 20 to 25 μm.

4.2.2 Flow Rate

Aluminium powder showed no free flow under gravity. On tapping it started flowing but stopped after a short time. While the composite powders were showing good flowability (Table 4.4). It was also observed that increasing the plating time and decreasing the bath load of aluminium powder increased the flow rate.

4.2.3 Apparent Density

Apparent density of Al-Ni composite powder was found to be lower than the aluminium powder. On increasing the plating time and decreasing the bath load of aluminium powder increased apparent density (Table 4.4).

4.2.4 Angle of Repose and Compressibility

Angle of repose for aluminium powder was found to be somewhat higher than the nickel coated aluminium powder (Table 4.4). Whereas there was not much difference in the compressibility of aluminium and Al-Ni composite powders (Table 4.4).

4.2.5 Differential Thermal and Thermogravimetric Analysis

DTA analysis of uncoated Al powder and two Al-Ni composite powders having 21 pct. and 35 pct. nickel showed the presence of an additional exothermic peak in composite powders at about $778^{\circ}C$ (Fig. 4.10). The amount of heat evolved indicated by the area under the peak, was more in the composite powder having higher nickel content.

TGA analysis showed that in both uncoated and nickel coated aluminium powders there was a weight loss of approx. 3 pct. to 3.6 pct. between $400-500^{\circ}C$.

4.2.6 X-ray Powder Diffraction Analysis

X-ray diffraction studies showed that Al-Ni composite powder peaks corresponding to aluminium and nickel (Table 4.5). Peaks for phosphorus could not be observed clearly because of interference from powder mounting material. The Al-Ni composite powder heated for 1 hour at $650^{\circ}C$ showed the presence of few extra peaks may correspond to Al_3Ni, NiO and Al_2O_3.

4.2.7 Al-Ni Composite Wire

Wires were prepared using Al-Ni composite powder
(Fig. 4.11). Crossection of these wires showed that there
was no large scale discontinuity. However, large amount of
microporosity was observed (Fig. 4.12).

4.3 DISCUSSION

During electroless nickel plating following reaction
occurs

$$Ni^{++} + 2H_2PO_2^- + 2H_2O \longrightarrow Ni + H_2 + 4H^+ + 2HPO_3^{--}$$

Hypophosphite ion   phosphite ion
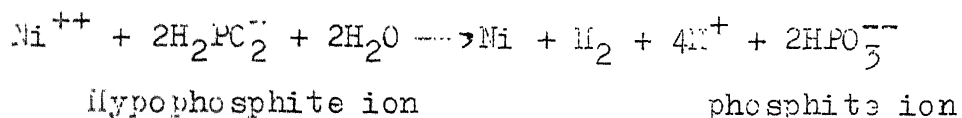
Therefore with increasing plating time a decrease in solution
pH, due to the formation of H^+ ions and an increase in
$Ni^{++}/H_2PO_2^-$ ratio in the solution due to higher consumption
of hypophosphite ions compared to nickel ions occurs. A
decrease in pH will reduce the efficiency of hypophosphite
ions (16) and will thereby tend to reduce the deposition
rate of nickel. An increase in $Ni^{++}/H_2PO_2^-$ ratio will also
decreases deposition rate of nickel (11, 17). In the present
investigation these two effects may explain decrease in
nickel deposition rate with increasing plating time.

The effect of increasing temperature on nickel
deposition rate is in agreement with results of other
investigators (18,19) who have reported that the rate of

deposition increases exponentially with temperature and spontaneous bath decomposition occurs at much higher temperatures.

Kurvyakova L.M. et al.[20] observed that the rate of electroless nickel deposition on iron powders increased with lowering the bath load of iron powder. However the percentage of nickel recovered from the solution decreases with decreasing bath load. Similar effect of bath load on nickel deposition rate has been observed in the present investigation.

The flow rate and apparent density of powders is effected (21) by

1. Powder shape, size and size distribution.
2. Specific gravity of the components.
3. The thickness of the coating.
4. The coefficient of inter-particle friction.
5. Friction between the particle and the wall of the orifice.
6. Packing arrangement of particles.

Therefore in the present case an increase in flow rate with increasing plating time and decreasing bath load may be due to an increase in nickel coating and particles become less angular.

A decrease in apparent density of Al-Ni composite powder as compared to aluminium powder may be due to a change

in particle shape, size and size distribution. Whereas an increase in apparent density with increasing plating time and decreasing bath load may be due to increasing amount of nickel in the composite powder.

Angle of repose is a measure of powder flowability, lower the angle of repose (22) greater is the powder flowability. Carstensen et al.[23] observed that angle of repose decreased with increasing particle diameter. In the present investigation decrease in angle of repose of Al-Ni composite powders as compared to uncoated aluminium powder may be due to an increase in powder diameter on nickel coating. However, change of plating time and bath load appear to have not much effect on angle of repose.

CHAPTER 5

CONCLUSIONS

Following conclusions can be made on the basis of present investigation.

1. Electroless plating method is quite suitable for coating nickel on aluminium powder. In a single coating operation the maximum amount of nickel in the composite powder was about 25 percent.

2. Using an alkaline plating bath containing Nickel chloride 30 g/l, Sodium hypophosphite 10 g/l, Sodium citrate 100 g/l, Ammonium chloride 50 g/l, optimum conditions for satisfactory coating of nickel on aluminium powder were found to be $90^{\circ}C$, pH 9.0, plating time 30 minutes and bath load 20 gm/l.

3. Flowability of Al-Ni composite powder is better than that of uncoated aluminium powder.

4. Using these powders it may be possible to produce wires which could possibly be flame sprayed in wire metallizing gun.

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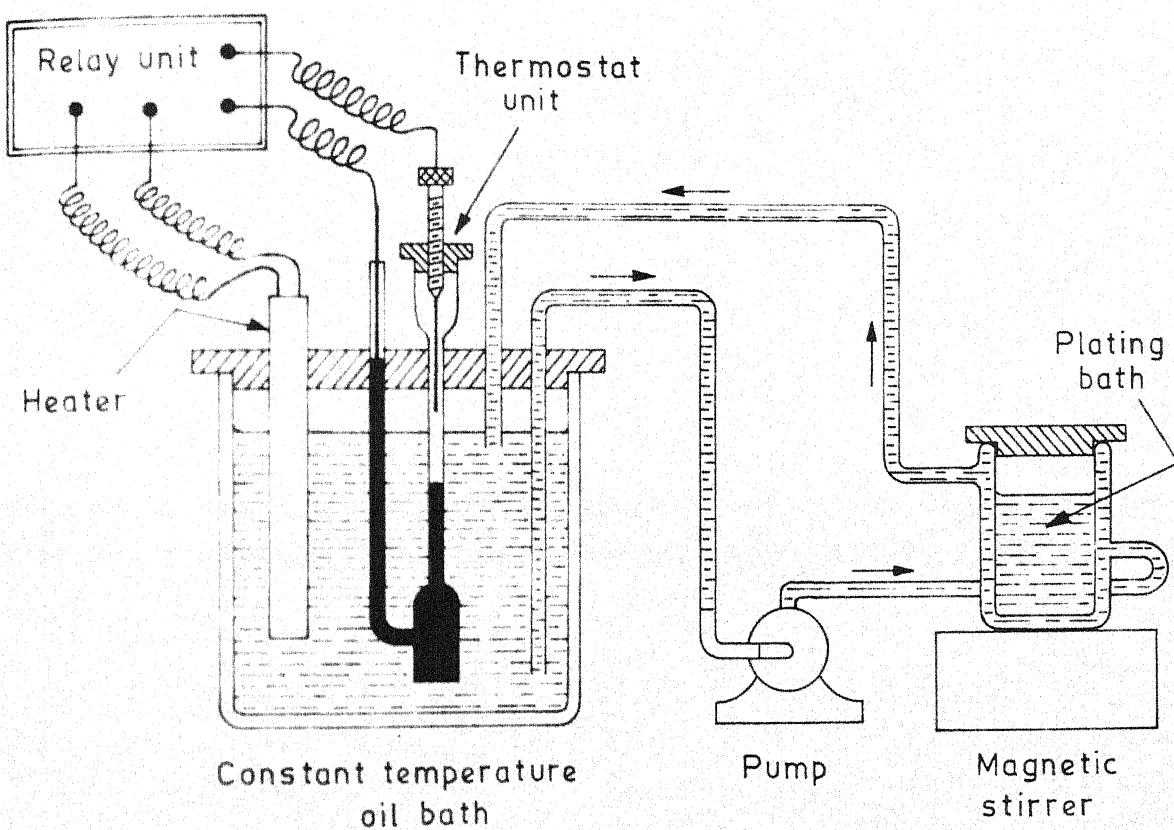   Technology, 15, 129-131 (1976).

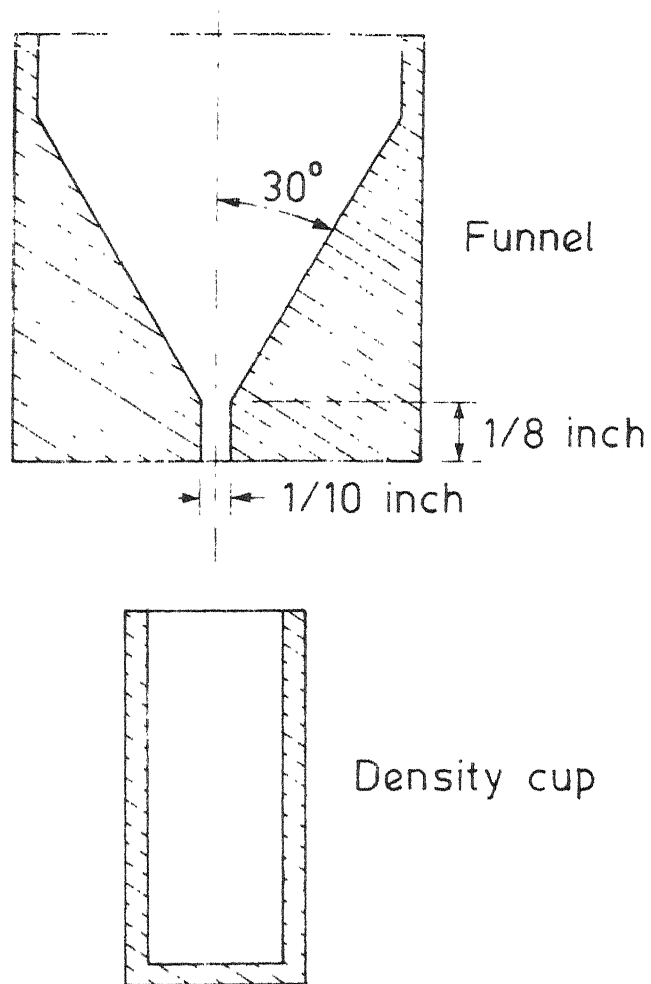Fig. 3.1   Experimental set-up (schematic)

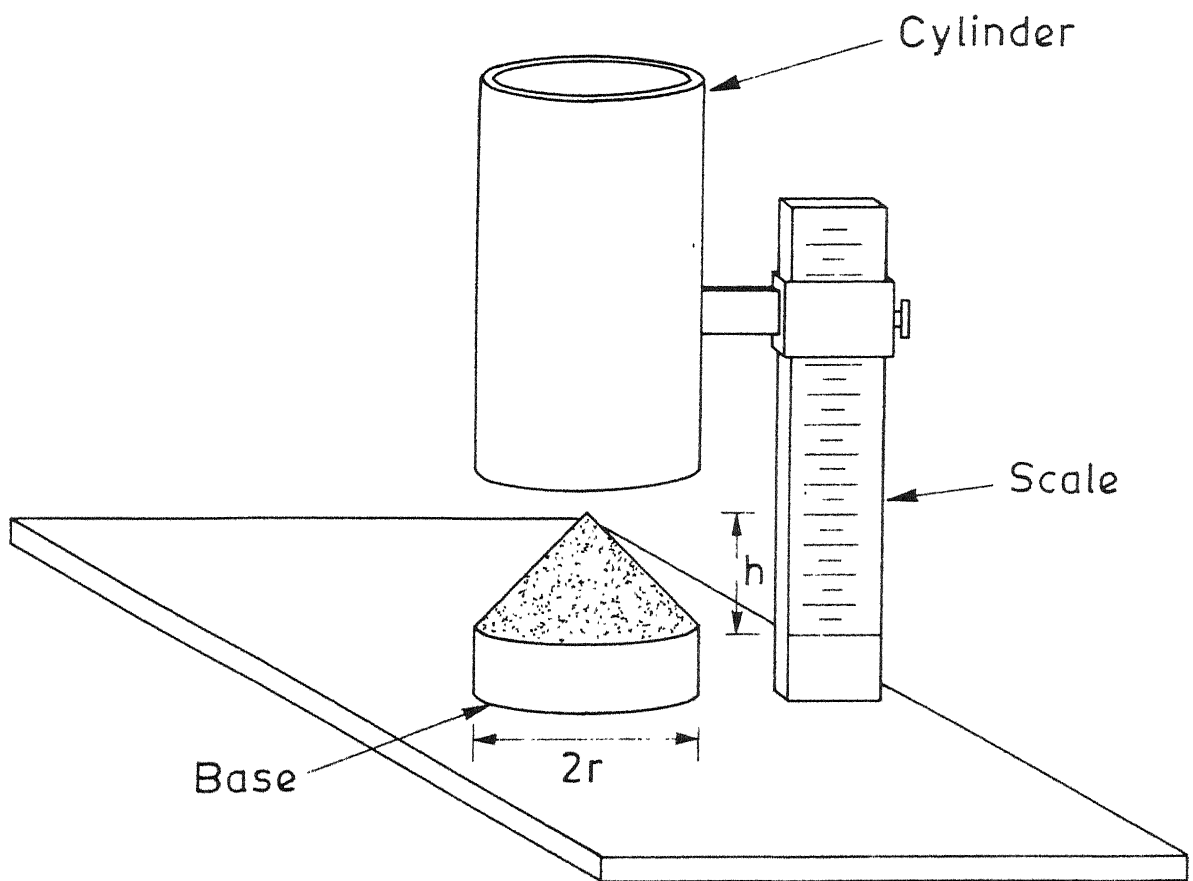Fig. 3.2 Flowmeter and apparent density
measuring cup.

Fig. 3.3   Measurement of angle of repose.

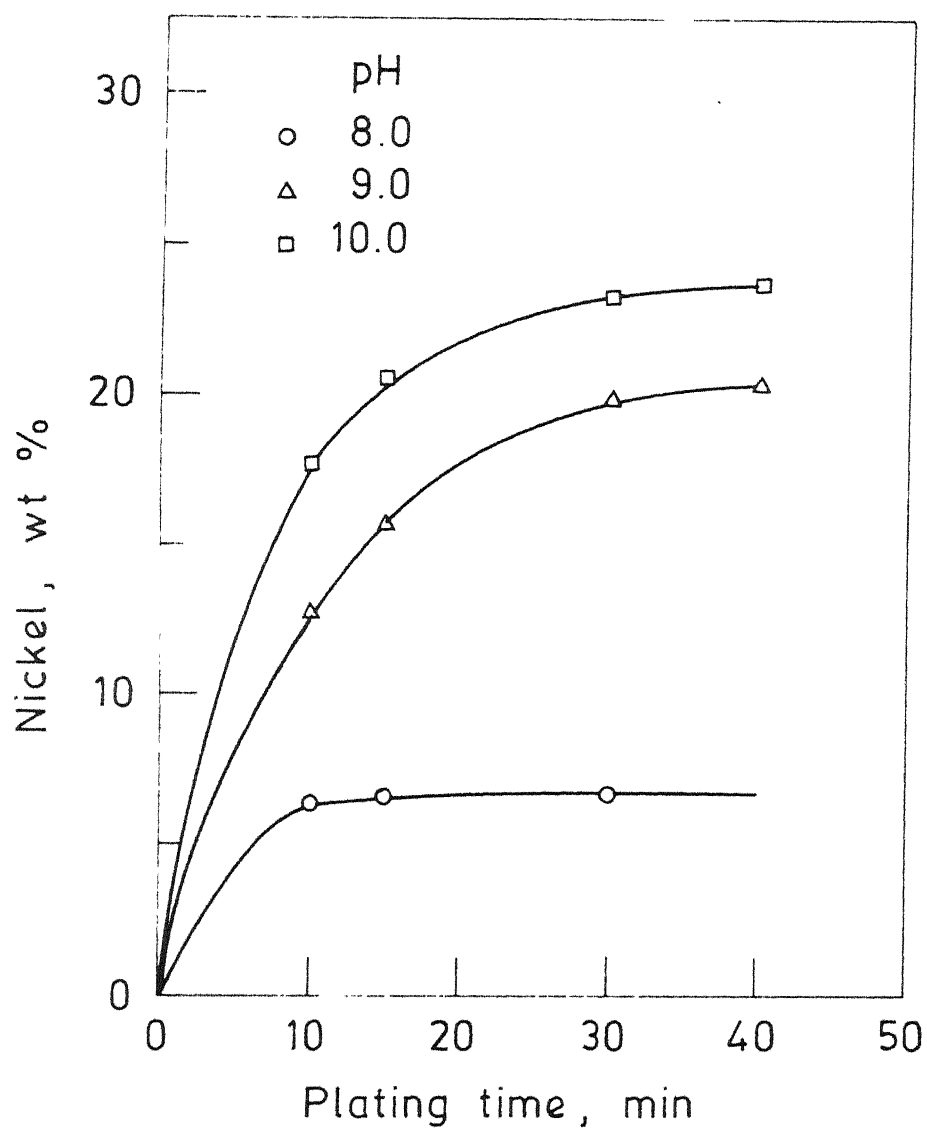Fig. 4.1 Effect of plating time at different
pH on nickel content of Al-Ni composite
powder at 85 ±1°C and 20 g/l bath
load.
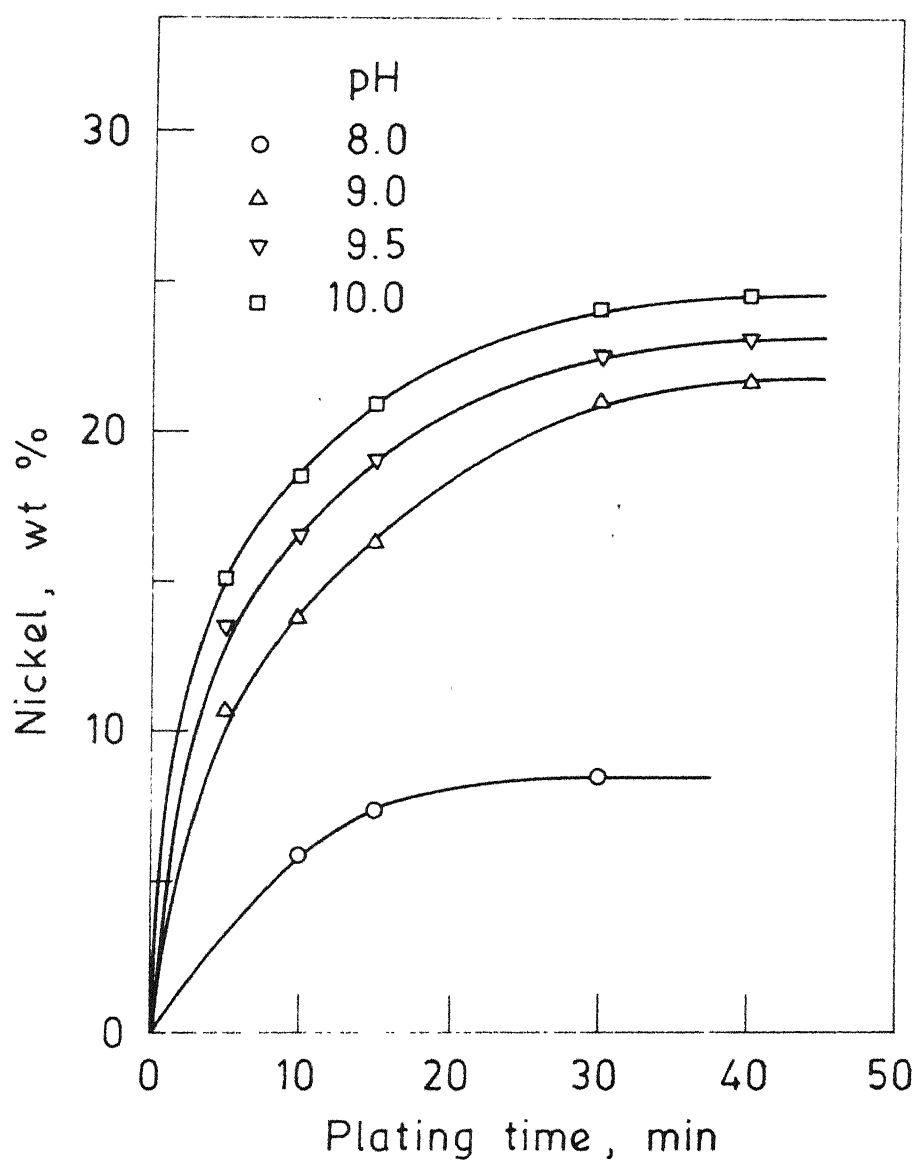
Fig. 4.2 Effect of plating time at different pH on nickel content of Al-Ni composite powder at 90 ± 1°C and 20 g/l bath load.
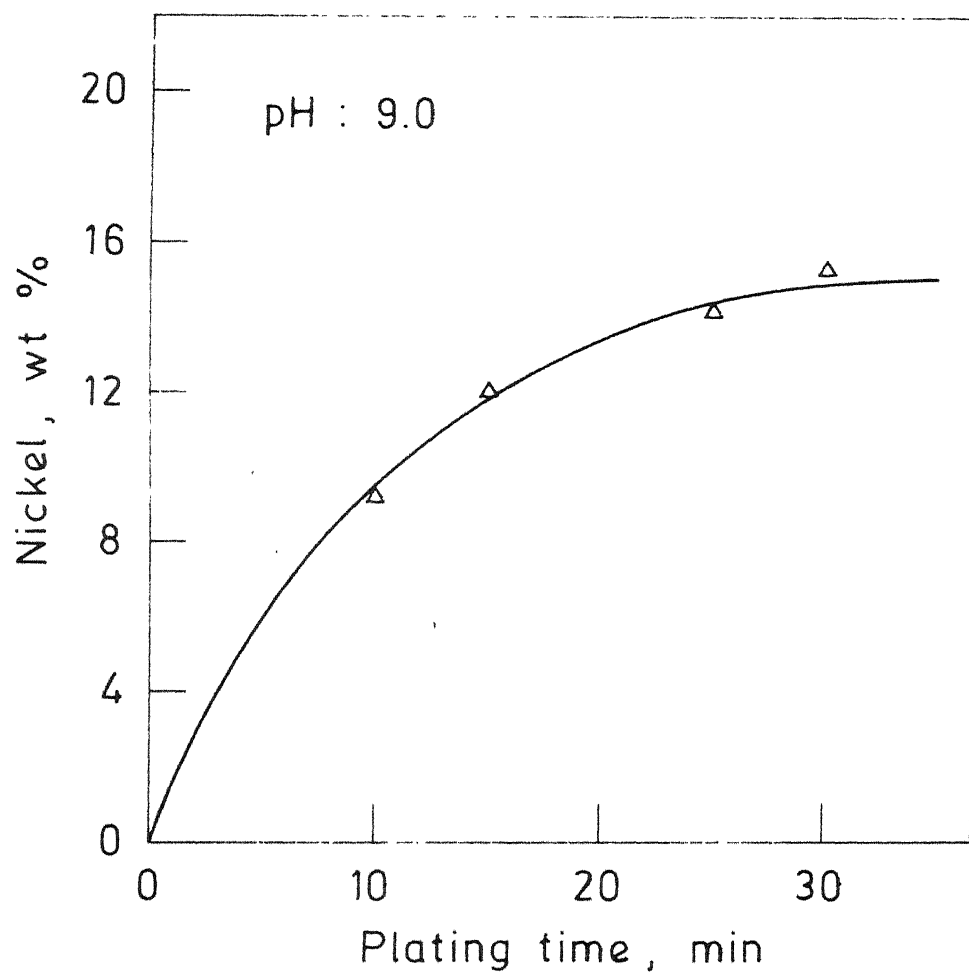
Fig. 4.3 Effect of plating time on nickel content of Al - Ni composite powder at 90 ± 1°C, pH 9.0, bath load 40 g/l.
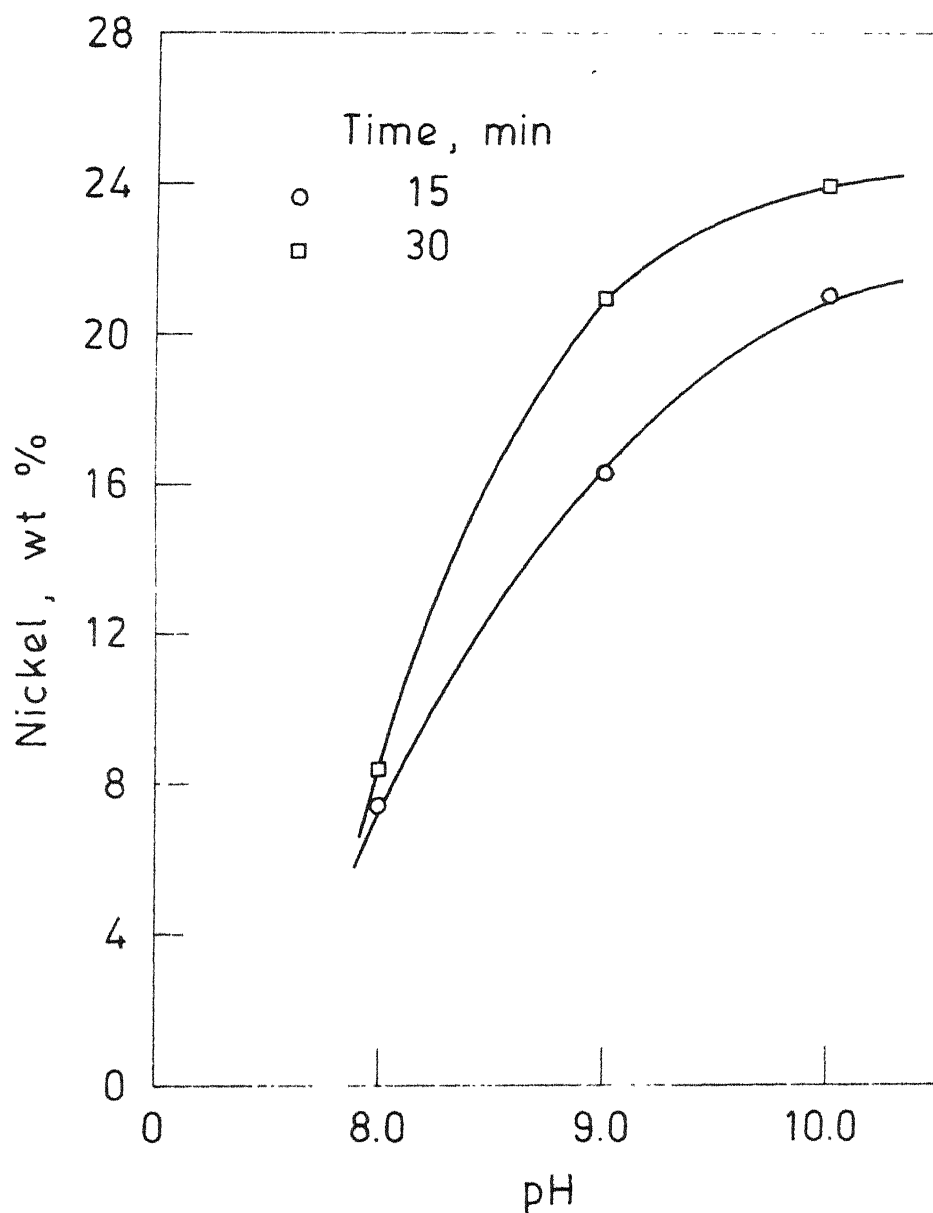
Fig. 4.4 Effect of pH at different plating time
on nickel content of Al - Ni composite
powder at 90 $\pm$ 1°C and 20 g/l bath
load .
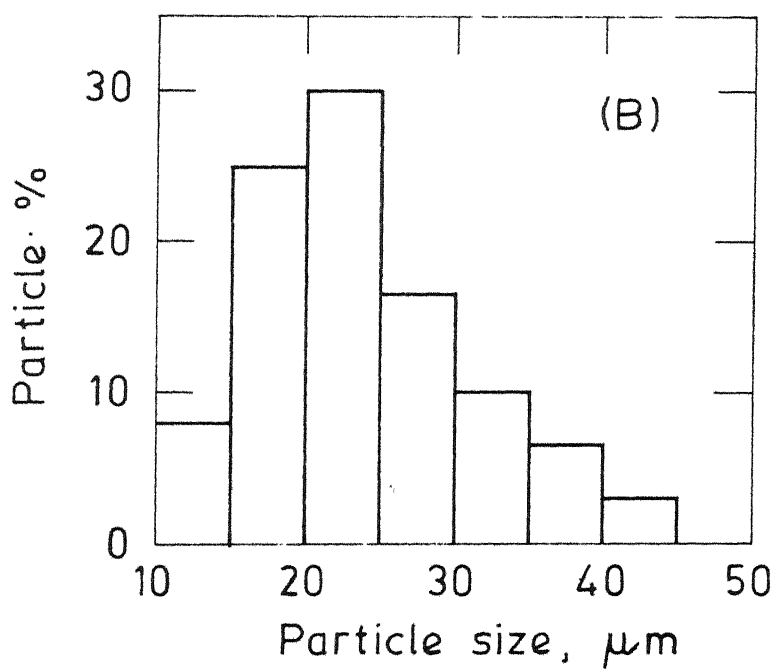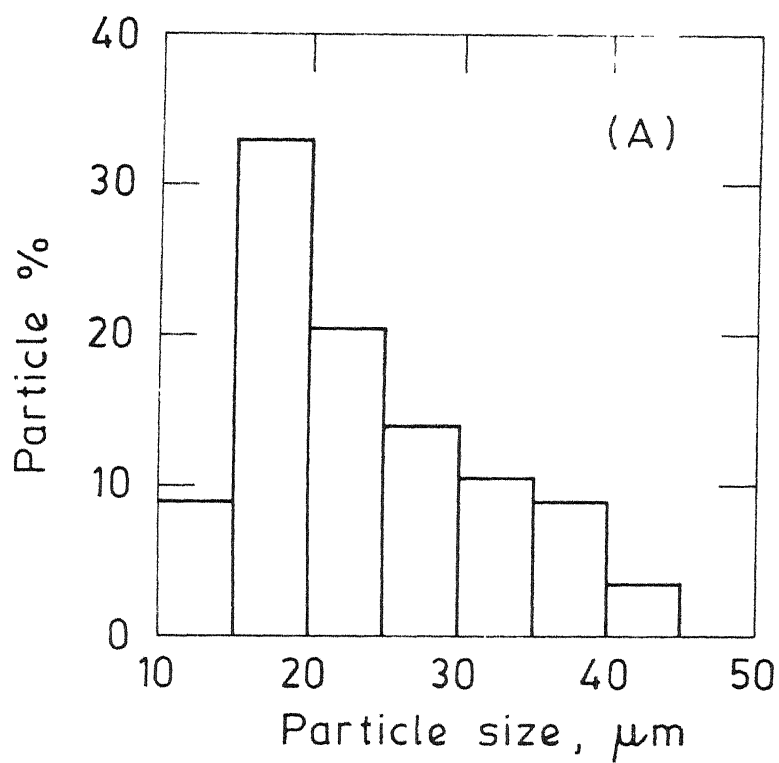
Fig. 4.5   Particle size distribution (A) Al powder,
(B) Al-Ni composite powder.
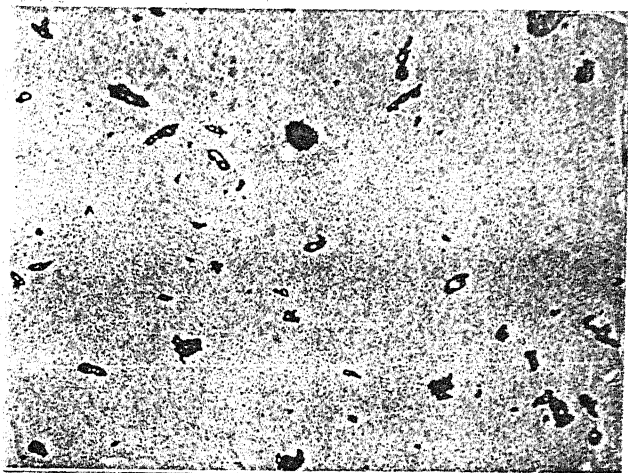
Fig. 4.6   Al Powder Particles
           X 200.
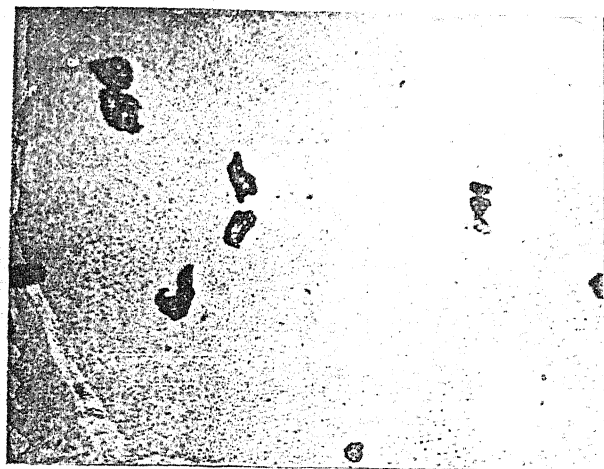
Fig. 4.7   Al-Ni Composite
           Powder Particles
           X 200.
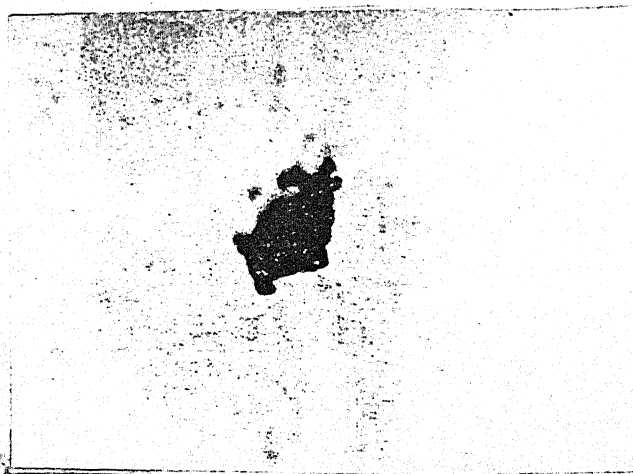
Fig. 4.8   Agglomerated Al-Ni
           Composite Powder
           X 400.
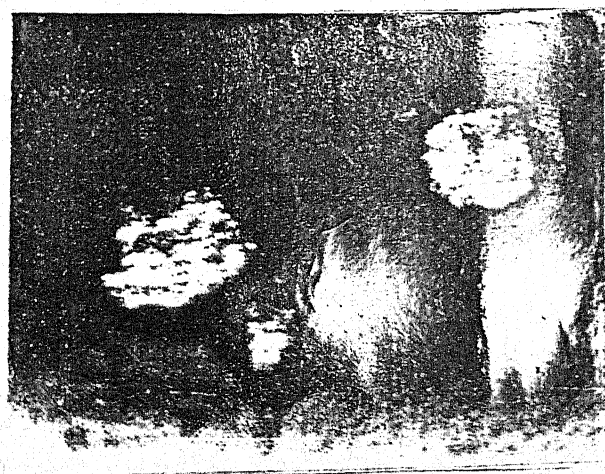
Fig. 4.9   Cross-section of
           Ni Coated Alumi-
           nium Particles
           X 800.
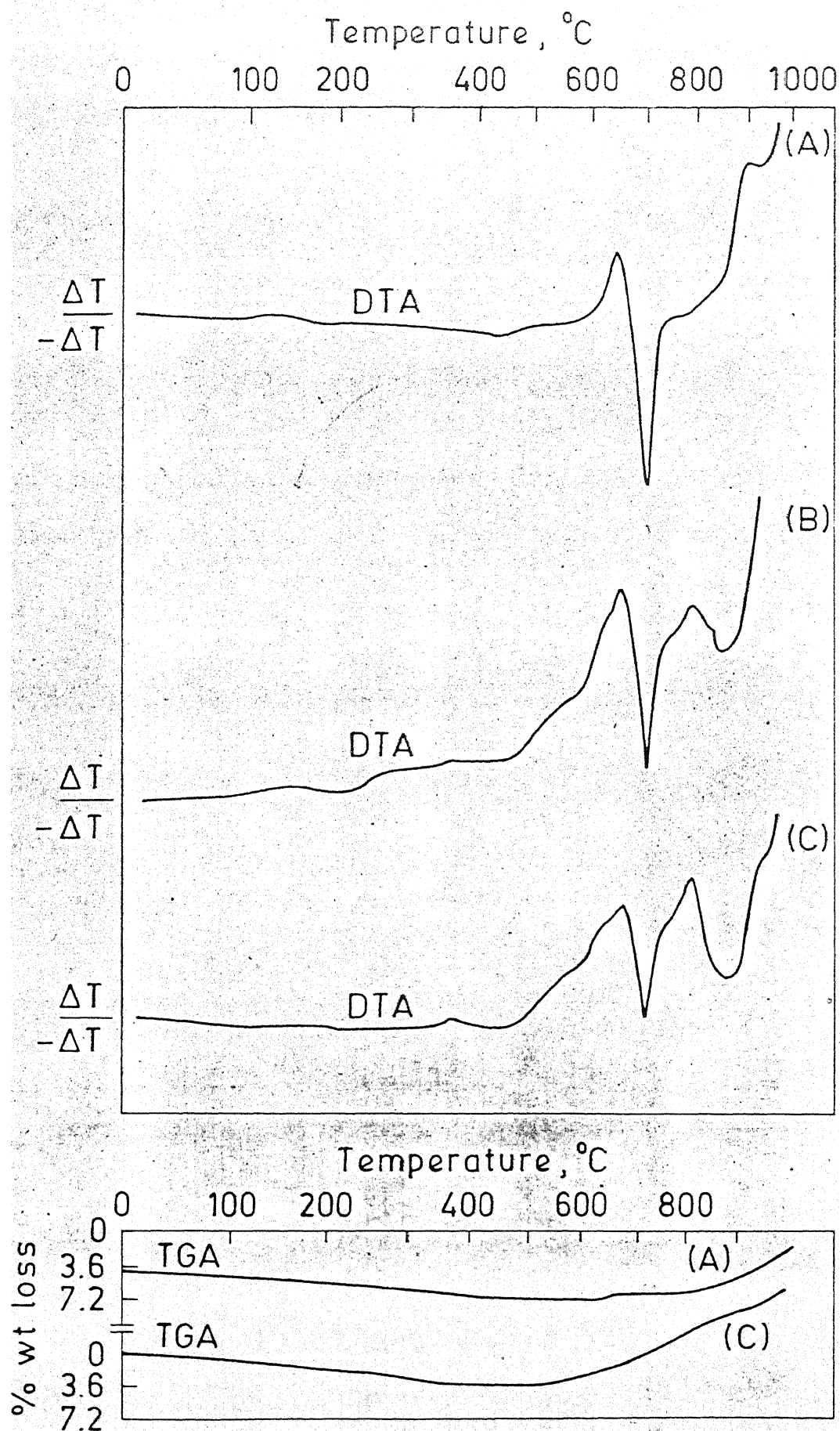
Fig. 4.10 DTA and TGA curves of (A) Al powder,(B) Al-Ni composite powder [Ni 21 wt %], (C) Al-Ni composite powder [Ni 35 wt %]
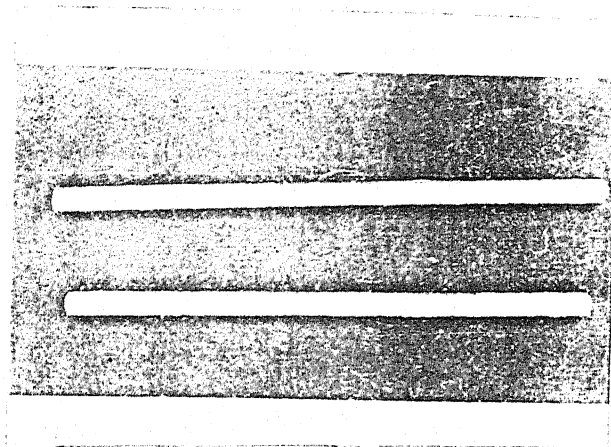
Fig. 4.11 Wire of Composite
Al-Ni Powder.
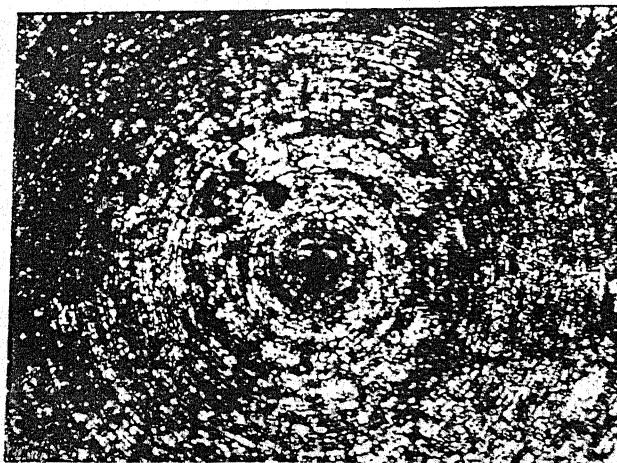
Fig. 4.12 Cross-section of
Al-Ni Wire X 200.

TABLE 3.1

ELECTROLESS NICKEL PLATING BATH

Constituents	Amount gm/l
Nickel chloride ($NiCl_2.6H_2O$)	30
Sodium hypophosphite ($NaH_2PO_2.H_2O$)	10
Sodium citrate ($Na_3C_6H_5O_7.2H_2O$)	100
Ammonium chloride (NH_4Cl)	50

pH   —   8-10

Temperature   — 90 $\pm$ 1°C

TABLE 4.1

EFFECT OF PLATING TIME ON NICKEL CONTENT OF Al-Ni
COMPOSITE POWDER AT DIFFERENT pH, $85 \pm 1^{\circ}$C, BATH
LOAD 20 gm/l

pH	Time (min)	Wt. powder dissolved (gm)	Ni-dmg ppt (gm)	Ni (gm)	Wt. pct. Ni
8.0	10	0.1976	0.0615	1.25×10^{-2}	6.32
8.0	15	0.2013	0.0640	1.30×10^{-2}	6.46
8.0	30	0.2019	0.0650	1.32×10^{-2}	6.54
9.0	10	0.2201	0.1395	2.83×10^{-2}	12.87
9.0	15	0.2004	0.1595	3.24×10^{-2}	15.95
9.0	30	0.2021	0.1960	3.98×10^{-2}	19.70
9.0	40	0.2021	0.2010	4.08×10^{-2}	20.20
10.0	10	0.2027	0.2070	4.20×10^{-2}	17.75
10.0	15	0.2137	0.2170	4.40×10^{-2}	20.60
10.0	30	0.2018	0.2315	4.70×10^{-2}	23.30
10.0	40	0.2079	0.2415	4.90×10^{-2}	23.60

TABLE 4.2

EFFECT OF PLATING TIME ON NICKEL CONTENT OF Al-Ni COMPOSITE
POWDER AT DIFFERENT pH, $90\pm1^{\circ}$C, BATH LOAD 20 gm/l

pH	Time (min)	Wt. powder dissolved (gm)	Ni-dmg ppt (gm)	Ni (gm)	Wt. pct. Ni
8.0	10	0.2022	0.0590	1.19×10^{-2}	5.93
8.0	15	0.1976	0.0715	1.45×10^{-2}	7.35
8.0	30	0.2013.	0.0837	1.70×10^{-2}	8.45
9.0	5	0.2008	0.1050	2.13×10^{-2}	10.62
9.0	10	0.1994	0.1350	2.74×10^{-2}	13.75
9.0	15	0.2004	0.1595	3.24×10^{-2}	16.15
9.0	30	0.2021	0.2105	4.09×10^{-2}	20.97
9.0	40	0.2032	0.2165	4.39×10^{-2}	21.59
9.5	5	0.2035	0.1355	2.75×10^{-2}	13.52
9.5	10	0.2007	0.1635	3.32×10^{-2}	16.55
9.5	15	0.2031	0.1895	3.85×10^{-2}	18.95
9.5	30	0.2029	0.2255	4.58×10^{-2}	22.57
9.5	40	0.2102	0.238	4.84×10^{-2}	23.0
10.0	5	0.2031	0.150	3.05×10^{-2}	15.0
10.0	10	0.2010	0.1820	3.69×10^{-2}	18.40
10.0	15	0.2049	0.2120	4.30×10^{-2}	21.02
10.0	30	0.2013	0.2385	4.86×10^{-2}	24.07
10.0	40	0.2123	0.2550	5.18×10^{-2}	24.38

TABLE 4.3

EFFECT OF PLATING TIME ON NICKEL CONTENT OF Al-Ni
COMPOSITE POWDER AT pH 9.0, $90\pm1^{\circ}$C, BATH LOAD 40 gm/l

pH	Time (min)	Wt. powder dissolved (gm)	Ni-dmg ppt (gm)	Ni (gm)	Wt. pct. Ni
9	10	0.1995	0.090	1.8×10^{-2}	9.14
9	15	0.2047	0.1205	2.44×10^{-2}	11.95
9	25	0.2110	0.1460	2.96×10^{-2}	14.06
9	30	0.1989	0.1480	3.0×10^{-2}	15.19

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TABLE 4.4

PHYSICAL PROPERTIES OF PURE Al AND COMPOSITE Al-Ni
POWDERS PRODUCED AT pH 9 AND TEMPERATURE $90 \pm 1^{\circ}C$

Powder code	Flow rate (sec/50 gm)	Apparent density gm/cc	Angle of repose	pct. compressibility
A	197.2	0.766	46.56°	22.53
B	195.3	0.80	46.60°	22.6
C	208.6	0.639	46.24°	22.9
D	199.2	0.709	45.95°	21.96
Al powder	No free flow	1.12	48.05°	23.0

A - bath load 20 gm/l, plating time 15 min

B - bath load 20 gm/l, plating time 30 min

C - bath load 40 gm/l, plating time 15 min

D - bath load 40 gm/l, plating time 30 min

TABLE 4.5

X-RAY DIFFRACTION ANALYSIS FOR PREPARED AND HEAT TREATED Al-Ni COMPOSITE POWDER. (Rad. CuK$_\alpha$, Filter Ni)

Al-Ni powder		Al-Ni (650°C/ hr)	
d(A$^\circ$)	I/I$_1$	d(A$^\circ$)	I/I$_1$
2.578	11.5	2.5815	12.9
2.3237	100.0	2.4044	50.4
2.2352	9.4	2.3324	100
2.0166	88.3	2.2352	6.8
1.7585	13.7	2.0840	59.5
1.4289	39.9	2.0208	68.7
1.3504	5.0	1.4754	29.7
1.2424	7.3	1.4308	41.2
1.2186	38.30	1.2563	13.7
1.1680	11.5	1.2177	41.9
1.0610	6.8	1.2043	10.3
1.0123	6.4	1.1686	12.9
		1.0130	7.3

APPENDIX

ASTM X-RAY DATA FOR Al, Ni, αAl_2O_3, NiO, Al_3Ni AND PHOSPHORUS

Al		Ni		$\alpha-Al_2O_3$		NiO		Al_3Ni		P	
$d(A^O)$	I/I_1	$d(A^O)$	I/I_1	$d(A^O)$	I/I_1	$d(A^O)$	I/I_1	$d(A^O)$	I/I_1	$d(A^O)$	I/I_1
2.338	100	2.034	100	3.479	75	2.410	91	4.02	40	4.23	100
2.024	47	1.762	42	2.552	90	2.088	100	3.44	100	3.67	70
1.431	22	1.246	21	2.379	40	1.476	57	3.01	60	3.28	70
1.221	24	1.062	20	2.085	100	1.259	16	2.71	3	2.59	70
1.169	7	1.017	7	1.740	45	1.206	13	2.55	30	2.32	50
1.0124	2	0.881	4	1.601	80	1.044	8	2.46	40		
0.9289	8	0.8084	14	1.514	6	0.958	7	2.40	20		
0.9055	8	0.788	15	1.510	8	0.9348	21	2.26	5		
0.8266	8			1.404	30	0.8537	17	2.18	70		
				1.374	50			2.16	80		
				1.276	4			2.07	100		
				1.239	16			2.01	40		
				1.189	8			2.00	90		
				1.147	6			1.97	70		
				1.1255	6			1.93	100		
				1.0988	8			1.88	30		
				1.0781	8			1.76	20		
				1.0426	14						
				0.9976	12						
				0.9076	14						
				0.9052	4						

Rad. CuK_{α_1}	Rad. CuK_{α_1}	Rad. CuK_{α_1}	Rad. CuK_{α_1}	Rad. CrK_{α}	Rad. CuK_{α}
Filter Ni	Filter Ni	Filter Ni	Filter Ni	Filter ?	Filter ?